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University of Delaware Center for Composite Materials
Composites Manufacturing Science Laboratory
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13. ABSTRACT (Maximum 200 words)
This program is a multidisciplinary research effort to develop an effective science base for the characterization and processing of composite and hybrid materials. The overall goal of the program is to provide useful results for the near-term optimization and control of established processing methods and extend the science base for relating processing to the behavior of composite materials. The research efforts are focused on the relationships of processing-induced microstructure to material behavior in the following projects: Processing and Microstructural Studies of Pre-Ceramic Based Composites; High-Strain-Rate Properties of Metal- and Ceramic-Matrix Composites; and Influence of Interphase Zones on the Behavior of Composite Materials and Bonding. These collective efforts are aimed at improving the reliability and extending the useful life of structural components. The research is augmented by a Scientific Exchange Program. The program is strongly supported and leveraged by ongoing projects in the Center's Industry/University Consortium program as well as by research projects initiated through strategic alliances with key industrial sponsors and Army laboratories. This multi-year program ended in January 1997, the projects have either been completed or transferred into either the Phase 2 program or the ARL CMR Program.

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Advani, S. G., "Mold Filling with Preforms," presented at the *UD–CCM/MSU Joint Composites Workshop on Manufacturing Science of Composites: Molding Processes*, University of Delaware, Newark, DE, September 20–21, 1995.

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* Note: List includes related publications/presentations and publications/presentations from leveraged programs.

(2) SCIENTIFIC PERSONNEL supported by this project and HONORS/AWARDS/ DEGREES received during this reporting period:

Principal Investigator
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Ian W. Hall, Associate Professor of Mechanical Engineering and Materials Science
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K. V. Steiner, Associate Scientist and Associate Director, UD–CCM
Jack R. Vinson, H. Fletcher Brown Professor of Mechanical Engineering

Graduate Students and Postdocs
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Dru Hartranft, Graduate Student, Department of Mechanical Engineering
Xiaoguang Huang, Postdoctoral Fellow
Anand Kalambur, Graduate Student, Materials Science Program
Jie Li, Postdoctoral Fellow
Hui-Ying Ma, Postdoctoral Fellow
Mark VanLandingham, Graduate Student, Materials Science Program
Metin Tanoglu, Graduate Student, Materials Science Program
Paul White, Postdoctoral Fellow

Degrees Granted
Annand T. Kalambur (M. MSE '96)
Thesis: Dynamic Compressive Mechanical behavior of a Silicon Carbide/Aluminum Metal-Matrix Composite
Advisor: Ian W. Hall

(3) Report of INVENTIONS (By TITLE ONLY):
None

(4) SCIENTIFIC PROGRESS AND ACCOMPLISHMENTS:
PROCESSING AND MICROSTRUCTURAL STUDIES OF PRE-CERAMIC BASED COMPOSITES
(A. Parvizi-Majidi and T-W. Chou)

Three types of SOLVAD™ preceramic polymers manufactured by Solvay, Germany, are being investigated as potentially useful polymers in the synthesis of silicon carbide. These are polycarbosilanes D-PPC [poly(diphenylcarbosilane), $(Ph_2SiCH_2)_n$]; D-PPMC 1 [poly(diphenyl-co-dimethyl) carbosilane), $\{(Ph_2SiCH_2)(Me_2SiCH_2)\}_n$] and D-PPMC 033 [$\{(Ph_2SiCH_2)(Me2SiCH_2)_3\}$n]. These polymers offer low viscosities and pyrolysis temperatures, making them attractive for composite,

coating, and binder applications. Their advantages, however, are tempered by their low ceramic yields. This investigation attempts to increase the ceramic yield and purity through control of the starting materials and processing conditions, including mixing, curing, and pyrolysis procedures. These polymers have been used to fabricate Nicalon™ fiber-reinforced SiC-based composites through multiple infiltration/pyrolysis cycles and SiC -based coatings on steel substrates. They have also been added as binders to fine SiC powder for the production of silicon carbide bodies. For the composite work, the polymers have been used with and without added mullite particles. All pyrolysis steps were performed in an argon atmosphere.

Modeling and Characterization of Through-the-Thickness Properties of Multi-Directionally Reinforced Textile Composites: Textile structural composites have received much attention for applications where improved through-the-thickness properties are required. However, the properties and mechanical behavior of these composites have not yet been adequately characterized, particularly in the thickness direction. The lack of experimental information is largely due to difficulty in testing these composites, which is caused by their coarse microstructures and large non-uniformities in local stresses and strains. This research investigates test methodologies for textile composites, particularly in the thickness direction. The experimental results will be used in combination with analytical prediction of the composite properties to provide a basis for the understanding of structure-property relationships of textile composites.

Mechanical Behavior of Thin-film Coating/Substrate Systems Under Nanoindentation: Experimental results have demonstrated the different failure mechanisms of material systems consist of hard-coating on soft substrate and soft-coating on hard substrate. An analytical model using Hankel's transform method is introduced to examine the displacement and stress fields of a thin-film coating/substrate system with perfect interfacial bonding under an axisymmetrical compressive loading on the coating surface. The present analysis can account for the influence of the film thickness and the material properties of the substrate. This knowledge of the stress fields provides the basis of understanding of the failure mechanisms of thin-film coating/substrate systems. This work has been transitioned into the ARL Composite Materials Research Program, Phase 2 (funding #DAAH04-95-2-0005).

HIGH STRAIN RATE PROPERTIES OF METAL- AND CERAMIC-MATRIX COMPOSITES (I. W. Hall, J. R. Vinson)

The MMCs investigated to date represent the three major classes of composites: continuous-fiber, discontinuous-fiber, and short-whisker- or particulate-reinforced composites. This research focuses on investigating the effect of high-strain-rate loading on the deformation and fracture properties and will be reported briefly for each type separately. Other results are also summarized.

Continuous-Fiber-Reinforced MMCs: Both quasi-static and high-strain-rate compression tests have been conducted on an A356 aluminum alloy reinforced with 35, 50, and 66 V_f% of M40 fibers. Two types of fiber conditions were investigated: in the first, fibers were not subjected to any surface treatment before incorporation into the matrix, while the second set of samples was prepared with fibers that had been subjected to an oxidative surface treatment. Cylindrical samples ~8 mm in diameter were tested in the longitudinal and transverse orientations at strain rates up to ~1700s^{-1}. Longitudinal specimens were found to exhibit strain-rate sensitivity. The average ultimate stress of quasi-statically tested samples was 288 MPa, while that of the high strain-rate samples was between 450 and 590 MPa. Transverse samples exhibited considerably less strain-rate sensitivity and much lower ultimate stresses, typically between 140 and 210 MPa. There was considerable scatter in the data for the longitudinal samples, much of which has been shown to arise from fiber misalignment with respect to the compression axis. Transverse samples showed very little scatter, and fractographic analysis in the scanning electron microscope showed these trends to be associated with the fracture modes. Transverse sample failure was dominated by the shear properties of the metal

matrix, and only localized fiber breakage occurred. Longitudinal samples, on the other hand, showed complex fracture modes in which the buckling of large fiber bundles was observed at the sample surface in contact with the anvils. This buckling led to the progressive longitudinal splitting of the sample.

Discontinuous-Fiber-Reinforced MMCs: This system comprises 12 to 25 V_f% of short Saffil (d-alumina) fibers in a matrix of Al-1%Cu, prepared by a squeeze casting route. The fiber architecture is planar random with the fibers lying preferentially in the plane perpendicular to the pressing direction. Tests have been conducted in two different directions—perpendicular and parallel to the pressing direction—which are considered to be anisotropic in terms of fiber alignment and residual stresses induced by fabrication. Samples have been mechanically tested at rates from quasi-static to $1500s^{-1}$ using the Split Hopkinson Pressure Bar, followed by extensive metallographic analysis. Data from a 15 V_f% sample tested parallel and perpendicular to the plane of preferred fiber orientation and data for an identically produced but unreinforced matrix alloy indicate that the presence of the fibers increases the yield stress significantly and that the effect is larger at higher strain rates. Also, the yield stress is highly strain-rate sensitive, with a transition occurring at strain rates in the region of $800\text{-}1200s^{-1}$. Fiber orientation has a clear effect on the mechanical properties. The metallographic analysis also includes investigation of the development of fiber fragmentation during testing. Samples are subjected to digestion of the matrix in dilute acid after testing, which leaves the fibers exposed for observation and quantitative analysis. It is found that fibers begin to fragment early in the test and are reduced to an average length of 20–50 mm, which corresponds closely to the critical length for these fibers. Thereafter, the composite ceases to behave like a fiber-reinforced composite and begins to behave instead like a conventional high-strength alloy.

Whisker-Reinforced MMCs: Samples containing 25 V_f% SiC whiskers were tested in compression at strain rates up to $\sim 1500s^{-1}$ and examined by optical microscopy as well as scanning and transmission electron microscopies. The composite showed a strain rate sensitivity of the yield stress that was more pronounced than that of the unreinforced matrix. Fracture occurred along well-defined shear planes, within which the reinforcing whiskers were pulverized.

SiC/Al_2O_3 CMCs: Samples of a SiC/Al_2O_3 /Al CMC (armor) were prepared and tested. The results to date show considerable scatter, but it is clear that higher strain rates lead to more extensive fragmentation of the specimens and, hence, greater energy absorption. In addition, the retained metallic aluminum between the SiC and Al_2O_3 grains undergoes extensive plastic deformation and may critically be involved in the energy absorption processes.

Modeling of the Mechanical Properties of Damaged Composites: Work has begun on developing a model to predict the residual mechanical properties of damaged composites. When an MMC is stressed, possibly to levels below the macroscopic yield stress, a variety of damage mechanisms may occur, including fiber debonding and fiber cracking. Much of the above work also confirms that fiber cracking is a common phenomenon during the early stages of plastic deformation, with the consequence that residual properties after mechanical working or impact loading may be impaired. This work will compare the predictions of the model with results from a series of experiments.

Dynamic Compressive Mechanical Behavior of a SiC_w/Al Metal-Matrix Composite: The high strength and stiffness, combined with the low density, of SiCw/Al metal-matrix composites (MMCs) makes these materials an ideal choice for aerospace applications. However, such applications may entail dynamic loading conditions, making an assessment of this behavior essential for a safe and economic design. The main aim of this research is to study the dynamic behavior of a 25% V_f SiC_w/2124 Al MMC under compressive loading. The experiments were carried out using the Split Hopkinson Pressure Bar (SHPB) at strain rates varying from $300s^{-1}$ to $1200s^{-1}$. The effect of specimen geometry was also investigated by varying the specimen aspect ratios. Quasi-static compression tests were performed using an Instron machine. Similar tests were conducted on the unreinforced matrix alloy (2024 Al) for comparison. The results clearly showed that the MMC exhibited

very little rate sensitivity in the range tested and was no different from the unreinforced alloy. The specimen geometry variation had no effect on the above result. The MMC failed at strain rates in excess of $700s^{-1}$ and during quasi-static loading. The failure was ductile and was caused by the coalescence of microvoids along the planes of maximum shear stress. The unreinforced alloy did not fail. The SHPB test consisted of compressively loading cylindrical specimens at strain rates varying from 300 to $1200s^{-1}$. The stress-strain data was extracted using strain gages and by using the theory of one-dimensional wave propagation in rods, as originally developed by Kolsky. After the tests, metallographic investigations were done on selected specimens using SEM and TEM.

Influence of Interphase Zones on the Behavior of Composite Materials and Bonding (R. L. McCullough, J. W. Gillespie Jr.)

A Liebold X-ray photoelectron spectroscopy (XPS) unit has been transferred from the Army lab at Watertown to UD–CCM. The unit has been refurbished for use by both UD–CCM and Army personnel. This instrument is crucial to the identification of reactive surface moieties on the surface of fibers. In addition, this instrument will be used to identify degradation mechanisms localized at interfaces and to characterize loci of failure for polymer interphases. A Digital Instruments atomic force microscope system has been acquired, and personnel have been trained in its operation. Research is underway to evaluate the AFM for use as a quantitative probe of the fiber-matrix interphase zone to detect property gradients.

Environmental Effects on Thermoset Durability—Interactions in the Interphase: Composites tend to degrade upon environmental exposure due to a number of factors. These include interactions with moisture, oxygen, solvents, radiation, and thermal cycling. Degradation can occur via multiple mechanisms, including hydrolysis or oxidation of the resin, plasticization of the resin (i.e., lowering of the Tg) and microcracking to relieve residual stresses. These mechanisms can be enhanced in the vicinity of the fiber surface due to interphase formation The interphase is the region between the fiber and the neat resin, and it can have properties differing from either. For example, molecular weight segregation of the resin occurs in the interphase due to the restricted movement of large molecules near the fiber surface. Local concentration variations can occur due to selective affinity (or repulsion) of one component by the surface. The extent of reaction can also be affected by the interphase. All of these effects change the local physical properties of the system, which may influence the system's response to environmental exposure. Using differential scanning calorimetry, it has been shown that the presence of the glass surface or an accelerator can alter the kinetics. Neat resin, Dow Derakane 411-C-50, was isothermally cured at 100°C, with 1.5% Trigonox 239A as an initiator. To demonstrate the effect of a glass surface, 1-mm glass beads were placed in the pan along with the resin, cured in the same manner. The kinetics could still be modeled using an autocatalytic model, but the extent of cure was reduced. With an accelerator, (cobalt napthenate, or CoNap), the neat resin system kinetics do not follow the autocatalytic model. These changes affect the morphology and stoichiometry of the resin/fiber interphase, which affects the uptake of moisture in the interphase.

Investigating the Interphase Using Atomic Force Microscopy: Atomic force microscopy (AFM) has become an extremely useful tool in materials science applications. In this work, the capabilities of the AFM are extended to measure changes in local elasticity over a sample surface. Through appropriate analysis of AFM force curves, much information can be obtained regarding the mechanical, chemical, and adhesive properties of the surface. To date, very little effort has been dedicated to examination of the contact portion of the force curve. This region contains valuable information regarding the nanoscale mechanical response of the sample. In this study, a technique is

developed that relates the elastic modulus of the sample to the sample response measured using AFM force curves.

Bonding: Bonding efforts have focused on the development of a novel bonding technology for dissimilar materials, Diffusion-Enhanced Adhesion, or DEA. In this work, the diffusion of a bisphenol A epoxy into as-received amorphous PEEK film and the associated morphological changes in PEEK due to this diffusion were investigated. The presence of diffusion at the epoxy/PEEK interface has been established through autoclave processing studies. Attenuated Total Reflectance Fourier Transform Infrared Spectroscopy (FTIR-ATR) studies have been used to characterize this diffusion mechanism and also to optimize the dwell time-temperature window for autoclave processing. The diffusion process was studied at various time intervals by measuring the change in absorbance bands of selected characteristic peaks in each polymer. It was concluded that the diffusion of epoxy into PEEK is anomalous, and a pseudo-diffusion coefficient was determined using the experimental data. Further, the presence of induced crystallinity in the PEEK film from epoxy diffusion was studied using wide-angle X-ray scattering techniques and identified as an important consideration for autoclave process optimization. This work has been extended through technology transfer to the United Defense Limited Partnership (UDLP)/TARDEC Composite Armored Vehicle (CAV) Program. Tests conducted by UDLP have shown that the DEA approach is the first adhesive to satisfy ballistic multi-hit performance requirements.

Cure Simulation and Residual Stress Predictions for Novel Integral Armor Concepts: An in-house cure simulation and residual stress predictive capability is developed based on integration of the commercially available finite element code ABAQUS and the pre- and post-processor PATRAN. This tool is used to predict and study the cure history and residual stress development within single-step-processed integral armor concepts. Both vinyl-ester and epoxy matrix composite systems are considered. Parametric studies focus on the effects of the ceramic core on thermal and degree-of-cure gradients as well as on residual stress development. Analytic solutions are also developed to validate predictions for the traditionally recognized thermal expansion mismatch induced residual stresses. Parametric studies indicate that the ceramic core does have a significant effect on the thermal and degree-of-cure distributions that develop within the part. From a processing standpoint, the presence of the core is advantageous, since it acts as a heat sink that reduces internal part temperatures, thermal and degree-of-cure gradients, and residual stresses. This investigation demonstrates that one-step curing is a viable processing technique for integral armor structures utilizing thick-section composites.

ARO/ARL Scientific Exchange Program (R. L. McCullough, K. V. Steiner)

Five ARL employees are currently in residence at the Center: Bruce K. Fink (Ph.D. MSE '91), Travis A. Bogetti (Ph.D. ME '90), Steven H. McKnight (Ph.D. MSE '96), William O. Ballata (M. ME'97), and Robert Klinger (M. MSE '99). They are collaborating with Center faculty, students, and staff in a variety of areas, including SMARTweave, dissimilar welding, thick-section mechanics, and SCRIMP modeling and processing.

(5) TECHNOLOGY TRANSFER

The ARL Composite Materials Research Program has effected technology transfer through the following general mechanisms:

- collaborative research
- equipment/facilities sharing
- publication in journals and presentation at conferences and symposia
- seminar series

- research focus groups
- co-advisorship of students
- software/simulation development

In addition, the following programs that provided leveraging for the ARL Composite Materials Research Program:

- Tuskegee University Research Consortium, Intelligent Resin Transfer Molding for Integral Armor Applications
- Composites Manufacturing Education and Training Program with Michigan State University
- Composite Armored Vehicle–Advanced Technology Demonstrator (CAV–ATD)
- DARPA Rapid Placement Technology for Advanced Composites Manufacturing (RAPTECH- ACM) Program
- Composite Army Bridge program
- SMARTweave Implementation at Northrup Grumman
- XM-194 Gun Mount Shield
- Lightweight body armor programs with Army and industry

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